ORIGINAL ARTICLE

# Synthesis, Characterization and Surface Activity of Alkyl *m*-Xylene Sulfonate Isomers

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**Abstract** This paper deals with the synthesis of a series of alkyl *m*-xylene sulfonate isomers (with the *m*-xylene located at the eighth carbon atom along the long alkyl chain) by the Friedel–Crafts reaction, and the Grignard reaction followed by hydrogenation. The structures were confirmed by <sup>1</sup>H NMR. All analytical methods indicated high levels of purity of the isomers with the eighth carbon atom at the long alkyl chain. The critical micelle concentration (CMC), surface tension and maximum surface excess concentration at the CMC and area per molecule at the interface were determined. As the long alkyl chains increased the surfactant molecule tends to pack closely at the gas–liquid interface. Accordingly, the CMC decreased, the adsorption density increased, and the surface tension reduction was strengthened.

**Keywords** Alkyl *m*-xylene sulfonate · Isomerization · Surface activity

# Introduction

Chemical flooding is an important method for enhanced oil recovery (EOR) [1, 2] and surfactants play an important

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role in this technique. When surfactant solutions are added to the reservoir, they can lower interfacial tension between oil and water, change the rock wettability and solubilization, thus contribute to EOR [3-7]. Most surfactants used in EOR are mixtures of different compounds, such as petroleum sulfonates and heavy alkyl benzene sulfonates, which are mainly composed of alkyl benzene sulfonates with different molecular weights and alkyl chain lengths. Because of their complex components and unidentified structures, it is difficult to elucidate the relationship between the structures and properties. This motivates researchers to synthesize the alkyl aryl sulfonates with definite structures. A considerable number of investigations have been reported on the synthesis and surface properties of alkylbenzene sulfonate with the definite structures. Baumgartner [8, 9] synthesized the isomers of dodecyl benzene sulfonate with the benzene ring attached at different positions on the dodecyl chain for the first time. The detergency and wetting properties were reported and related with different structures and much work was focused on the foaming, wetting, surface tension. Doe [10, 11] and Wade [12] studied the interfacial tension between *n*-alkane and an aqueous solution of hexadecyl benzene sulfonate isomers with the benzene ring located from the 2nd to the 8th carbon atom, i.e., from 2-phenyl hexadecane sulfonate to 8-phenyl hexadecane sulfonate, and they characterized the surfactants by the concept of the preferred alkane carbon number  $(n_{\min})$  to attain minimum tension. As the benzene ring point of attachment is shifted from the extreme to the center of the *n*-hexadecane chain, the surfactant becomes more hydrophobic, but it is also more water soluble, which is an apparently paradoxical association of properties. In recent years, Yang [13, 14] and Zhao [15] synthesized a series of the hexadecyl benzene sulfonates. Zhang [16] synthesized multi-n-alkyl benzene

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sulfonates and reported characteristic parameters such as the critical micellar concentration (CMC), and the surface and interfacial tension at CMC. Qu [17] synthesized a series of isomers of hexadecyl *o*-xylene sulfonates and reported on some of their properties. However, the synthesis and surface properties of alkyl *m*-xylene sulfonate isomers where the aryl located at the eighth carbon atom along the long alkyl chain have not been reported.

In this paper we present the synthesis of a series of isomers of alkyl *m*-xylene sulfonate and we report their properties. The correct name of the sulfonate final product in which the sulfonated aryl ring group is located at the eighth carbon atom of long alkyl chain could be n-pentadecane (or *n*-hexadecane, or *n*-heptadecane) 8-*m*-xylene sulfonate and it is abbreviated in what follows as Cx-8MXS. The same kind of abbreviation is used for the other isomers, with the number "x" indicating the total number of carbon atoms in the alkyl chain. The routes to attain the synthesized compounds are illustrated in Scheme 1. The details are discussed later in the experimental section. Because of the steric hindrance, the acylation cannot take place in the ortho position of one of the methyl groups of the aryl ring; as a consequence, there is only one possible final isomer of the alkyl *m*-xylene sulfonate. It means that the only isomeric variation is due to the attachment position of the *m*-xylene on the long alkyl chain.

# **Experimental Procedures**

# Materials

The purity of all the chemicals being used was high grade. Caprylyl chloride, nonanoyl chloride, and decanoyl chloride were from Shalong (Huzhou Zhejiang Province China). *n*-Bromoheptane were from Yancheng (Jiangsu Province China). *m*-Xylene, ethyl ether, magnesium strips, anhydrous calcium chloride and sodium hydroxide were obtained from Kemiou (Tian Jin China). All reagents were distilled prior to use. Pd/C catalyst (5 %) was provided by the Dalian Institute of Chemical Physics (Dalian China). Hydrogen and nitrogen were of high grade purity from XueLong (99.99 % purity, Daqing China). <sup>1</sup>H-NMR spectra were obtained with the Bruker FT-300 spectrometer using CDCl<sub>3</sub> as solvent. Chemical shifts (d) were reported in ppm related to internal tetramethylsilane. After some optimization trials, the alkyl *m*-xylene sulfonate isomers (C15-8MXS, C16-8MXS, C17-8MXS) was carried out through the following steps [18].

#### Synthesis of Ketones

# 1-(2,4-Dimethyl-phenyl)-1-octanone (1a)

Octanoyl chloride (0.10 mol, 16.2 g) was added dropwise to a stirred solution of anhydrous AlCl<sub>3</sub> (0.13 mol, 17.36 g) and *m*-xylene (0.40 mol, 42.4 g), the mixture was cooled in an ice-water bath for 30 min and then heated at a reflux for 3 h. The mixture was then cooled and decomposed with ice-cooled dilute hydrochloric acid (100 mL). After the phase of separation, the upper layer was washed with a NaHCO<sub>3</sub> solution and deionized water until it became neutral, and finally dried over anhydrous calcium chloride. The excess of *m*-xylene was removed by distillation and the solvent was evaporated under reduced pressure. Compound **1a** was obtained with a 85.31 % yield.

### 1-(2,4-Dimethyl-phenyl)-1-nonanone (2a)

Nonanoyl chloride (0.10 mol, 17.6 g) was added dropwise to a stirred solution of anhydrous  $AlCl_3$  (0.13 mol, 17.36 g) and *m*-xylene (0.40 mol, 42.4 g), compound **2a** was obtained with a 83.52 % yield as yellow oil starting from following the same procedure described for **1a**.

#### 1-(2,4-Dimethyl-phenyl)-1-decanone (3a)

Decanoyl chloride (0.10 mol, 19.0 g) was added dropwise to a stirred solution of anhydrous  $AlCl_3$  (0.13 mol, 17.36 g) and *m*-xylene (0.40 mol, 42.4 g), compound **3a** was obtained with a 82.93 % yield as yellow oil starting from following the same procedure described for **1a**.

#### Synthesis of Alcohols

### 8-(2,4-Dimethyl)phenyl-pentadecyl-8-alcohol (1b)

The reaction was conducted under nitrogen. Traces of iodine were used to initiate the Grignard reaction. *n*-bromoheptane (0.1 mol, 17.91 g) was added dropwise to the mixture of the magnesium strips (50.4 g, 2.1 mol) and Et2O (50 mL), then the reaction was placed in reflux until the magnesium strips had disappeared. A suspension of **1a** (0.05 mol, 11.58 g) in Et2O (50 mL) was added dropwise over 30 min to the Grignard reagent and heated at reflux for 2 h. The resulting mixture was decomposed by ice-cold dilute hydrochloric acid (100 mL). After the separation phase, the aqueous solution was extracted three times with ether. All extracts were combined, washed with deionized water until neutral and dried over anhydrous calcium chloride. Then **1b** was obtained with a 85.31 % yield.

# 8-(2,4-Dimethyl)phenyl-hexadecyl-8-alcohol (2b)

*n*-Bromoheptane (0.1 mol, 17.91 g) was added dropwise to the mixture of the magnesium strips (50.4 g, 2.1 mol) and Et2O (50 mL), then the reaction was placed in reflux until the magnesium strips had disappeared. A suspension of **1a** (0.05 mol, 12.28 g) in Et2O (50 mL) was added dropwise over 30 min to the Grignard reagent and heated at reflux for 2 h. Compound **2b** was obtained with a 89.78 % yield as yellow oil starting from following the same procedure described for **1b**.

#### 8-(2,4-Dimethyl)phenyl-heptadecyl-8-alcohol (3b)

*n*-Bromoheptane (0.1 mol, 17.91 g) was added dropwise to the mixture of the magnesium strips (50.4 g, 2.1 mol) and  $Et_2O$  (50 mL), then the reaction was placed in reflux until the magnesium strips had disappeared. A suspension of **1a** (0.05 mol, 12.98 g) in Et2O (50 mL) was added dropwise over 30 min to the Grignard reagent and heated at reflux for 2 h. Compound **3b** was obtained with a 89.78 % yield as yellow oil starting from following the same procedure described for **1b**.

# Synthesis of Alkyl m-Xylene Isomers

#### 8-(2,4-Dimethyl)phenyl-pentadecane (1c)

The alkyl *m*-xylene isomers were synthesized by hydrogenation of the corresponding **1b** (0.1 mol, 33.3 g) in glacial acetic acid (50 mL) and perchloric acid (1.0 mL) using 1.0 MPa H<sub>2</sub> and 1.0 g Pd/C (5 %) catalysts. Pd/C was added in the high-pressure reactor for 12 h reaction until the pressure stabilized. Then the catalyst was filtered off and washed with petroleum ether. The filtrate was then decomposed with water. The aqueous solution was extracted twice with petroleum ether. The solvent was washed with NaHCO<sub>3</sub> solution and distilled water, respectively, until neutral, and dried over anhydrous calcium chloride. The alkyl *m*-xylene isomers were obtained after distillation under reduced pressure. Compound **1c** was obtained with a 93.24 % yield.

#### 8-(2,4-Dimethyl)phenyl-hexadecane (2c)

**2b** (0.05 mol, 17.35 g) in glacial acetic acid (50 mL) and perchloric acid (1.0 mL) using 1.0 MPa H<sub>2</sub> and 1.0 g Pd/C (5%) catalysts Pd/C was added in high-pressure reactor reaction 12 h until the pressure stabilized. Compound **2c** was obtained with a 95.66% yield as a yellow oil starting from following the same procedure described for **1c**.

# 8-(2,4-Dimethyl)phenyl-heptadecane (3c)

**3b** (0.05 mol, 18.05 g) in glacial acetic acid (50 mL) and perchloric acid (1.0 mL) using 1.0 MPa H<sub>2</sub> and 1.0 g Pd/C (5 %) catalysts Pd/C was added in high-pressure reactor reaction 12 h until the pressure stabilized. Compound **3c** was obtained with a 96.12 % yield as yellow oil starting from following the same procedure described for **1c**.

### Synthesis of Alkyl m-Xylene Sulfonates Isomers

# 2,4-Dimethyl-5-(1-heptyl)octyl Sodium Phenyl Sulfonate (1d)

Chlorosulfonic acid (0.05 mol, 5.53 g) was added dropwise to the **1c** (0.05 mol, 15.8 g). The reaction proceeded for 2 h at 10 °C. The sulfonation product was neutralized with a sodium hydroxide solution. After phase separation, the mixture was recrystallized three times with anhydrous ethanol. The final product was filtered and dried in a vacuum oven at 80 °C. The purity of the product was determined by the two-phase titration methods. Purity: 98.8 %. The structure was confirmed by <sup>1</sup>H NMR.

1d <sup>1</sup>H NMR [300 MHz, (CDCl<sub>3</sub>)]  $\delta = 0.80-0.88$ (t, -CH<sub>3</sub>, 6H), 1.06-1.22(b, -CH<sub>2</sub>-, 20H), 1.42-1.52 (t, Me-CH<sub>2</sub>-, 4H), 2.74(m, -CH-, 1H), 2.18-2.28(s, Ar-H<sub>1</sub>, 6H), 7.64(s, Ar-H<sub>2</sub>, 1H).

# 2,4-Dimethyl-5-(1-octyl)octyl Sodium Phenyl Sulfonate (2d)

Chlorosulfonic acid (0.05 mol, 5.53 g) was added dropwise to the 2c (0.05 mol, 16.5 g) the reaction proceeded for 2 h at 10 °C as the procedure described for 1d. The purity of

**2d** was determined by the two-phase titration methods. Purity: 98.3 %. The structure was confirmed by 1H NMR.

**2d** <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) $\delta$ :0.762–0.831(t, –CH<sub>3</sub> 1, 3H), 0.841–0.868(t, –CH<sub>3</sub> 2,3H), 1.072–1.145(b, –CH2–, 22H), 1.482(m, Me–CH2–, 4H), 1.900(s, Ar–CH<sub>3</sub> 1, 3H), 2.128–2.160 (s, Ar–CH<sub>3</sub> 2, 3H), 2.314–2.369(m, –CH–, 1H), 6.985(s, Ar–H<sub>1</sub>, 1H), 7.555(s, Ar–H<sub>2</sub>, 1H).

# 2,4-Dimethyl-5-(1-octyl)nonyl Sodium Phenyl Sulfonate (3d)

Chlorosulfonic acid(0.05 mol, 5.53 g) was added dropwise to the **3c** (0.05 mol, 17.2 g). The reaction proceeded for 2 h at 10 °C as the procedure described for **1d**. The purity of **3d** was determined by the two-phase titration methods. Purity: 98.9 %. The structure was confirmed by <sup>1</sup>H NMR.

**3d** <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) $\delta$  = 0.853(t, -CH<sub>3</sub>, 6H),1.202(b, -CH<sub>2</sub>-, 24H),1.468(t, Me-CH<sub>2</sub>-, 4H), 2.289(s, Ar-CH<sub>3</sub>, 6H), 2.692(m, -CH-, 1H), 6.751(s, Ar-H<sub>1</sub>, 1H), 7.635(s, Ar-H<sub>2</sub>, 1H).

The structures of **1d**, **2d**, **3d**were confirmed by <sup>1</sup>H-NMR spectral analyses. All analytical methods indicated a high level of purity of all the isomers.

### **Results and Discussion**

Properties and Purity of Intermediates and Final Products

Alkyl *m*-xylene sulfonates isomers were prepared following Scheme 1. The boiling points of the intermediates are listed in Table 1. It can be seen that the boiling point increased for the alkyl chain longer. And from ketone to alkane, the boiling point becomes lower. The purities of the Cx-MXS isomers was determined by two-phase titration methods. C15-8MXS, C16-8MXS, C17-8MXS recrystallized three times and their purities were 98.8, 98.3, 98.9 % respectively.

Surface Tension Studies of Different Alkyl *m*-Xylene Sulfonate isomers

The surface tension was measured by the drop-volume technique in a water bath with thermostat control at 298  $\pm$  0.1 K. The time scale for one drop was 3–50 min depending on the time required to attain equilibrium. The surface tension was then measured three times for each sample with a 3–50 min interval between each reading to ensure equilibrium data. The surface tension (c) is given by  $\gamma = Fmg/R = \varphi F V \Delta \rho/R$ , where *R* is the tip's radius,  $\Delta \rho$  the difference of the two phases, *g* the local gravity acceleration, *V* the volume of one drop,  $F(R/V)^{1/3}$  is a



$R_1$	$R_2$	Boiling stage (°C)	Pressure (MPa)		
Ketone					
C7H15	C7H15	247-251	0.088		
C <sub>8</sub> H <sub>15</sub>	C7H15	252–255	0.088		
C <sub>9</sub> H <sub>19</sub>	$C_7H_{15}$	257–258	0.094		
Alcohol					
C <sub>7</sub> H <sub>15</sub>	$C_7H_{15}$	240-242	0.086		
C <sub>8</sub> H <sub>15</sub>	$C_7H_{15}$	310-314	0.078		
C <sub>9</sub> H <sub>19</sub>	$C_7H_{15}$	240-265	0.093		
Alkane					
C7H15	C7H15	223–225	0.086		
C <sub>8</sub> H <sub>15</sub>	C7H15	284–286	0.078		
C9H19	$C_{7}H_{15}$	214–227	0.092		



Fig. 1 Surface tension versus aqueous molar concentration (log scale) of synthesized Cx-MXS isomers

correction factor which accounts for the nonsphericity of the drop. Each surface tension value was determined from the average at least five measurements.

Figure 1 shows the plot of surface tension versus concentration (in a log scale) of alkyl *m*-xylene sulfonates isomers, exhibiting a break point which correspond to a well-defined CMC beyond which remains essentially constant. As the long alkyl chains increased, the *m*-xylene ring located at the eighth carbon atom along the long alkyl chains. The surfactant molecule tends to pack at the gasliquid interface closely. This is because the surfactant molecules hydrophobic chain crimp together at the gasliquid interface. Accordingly, the CMC decreased, the adsorption density increases, and the tension reduction is strengthened.

Table 2  $\gamma_{CMC}$  and CMC values of Cx-8MXS

Surfactant	$\begin{array}{c} \text{CMC} \\ (10^{-5} \\ \text{mol } \text{L}^{-1}) \end{array}$	$\gamma_{CMC} \ (mN \ m^{-1})$	$ \begin{array}{c} \Gamma_{max} \\ (10^{-10} \text{ mol} \\ \text{cm}^{-2}) \end{array} $	A (nm <sup>2</sup> )	pC <sub>20</sub>
C15-8MXS	7.98	25.9	0.911	1.82	6.68
C16-8MXS	4.32	26.03	1.43	1.16	5.95
C17-8MXS	2.45	26.61	1.84	0.90	5.84

Table 3 Variation of CMC compared with the Rosen equation

Carbon atom number of the	15	16	17
hydrophobic chain			
CMC calculated by the Rosen expression	-2.82	-3.114	-3.408
CMC value of the fitting equation	-4.1004	-4.3573	-4.6142

Area per Molecule and Efficiency of Adsorption

The surface excess,  $\Gamma$  at the air–water interface was calculated by applying the Gibbs adsorption isotherm equation. The area per molecule at the interface was estimated from the corresponding value of  $\Gamma$ . Table 2 summarized the values of CMC,  $\gamma_{CMC}$ ,  $\Gamma_{max}$ ,  $A_{min}$  and pC20. As the long alkyl chains increases, the surfactant molecule tends to be packed more closely at the gas–liquid interface. Accordingly, the CMC decreases,  $\gamma_{CMC}$  increases, the surface area per molecule becomes smaller, and  $\Gamma_{max}$ increases, and the branched hydrophobic chain tends to crimp together at the gas–liquid interface.

A convenient measurement of the efficiency of adsorption at the gas–liquid interface is the pC20 parameter introduced by Rosen [19], which is the negative of the logarithm of the surfactant concentration in the bulk phase required to produce a 20 mN m<sup>-1</sup> reduction in the surface tension of the solvent. As shown in Table 3, pC20 decreases as the alkyl chains increased. The results indicate that energetically speaking, the increase in branching of the hydrophobic chain inhibits the transfer of a surfactant molecule from the bulk to the interface.

The CMC is a strong function of the length of the alkyl chain. Rosen [19, 20] reported that the variation of the CMC with the number of carbons in the hydrophobic tail group (*m*) can often be empirically described by the equation  $\text{Log}(\text{CMC}) = A + B \times m$ , where *m* is the carbon atom number of the hydrophobic chain, *B* is an empirical constant, and *A* is a constant for the particular temperature and homologous series, which may be determined from a known value of the CMC. For the alkyl *m*-xylene sulfonate  $\text{Log}(\text{CMC}) = -0.2469 - 0.2569 \times m$ , this means that for the homologue with the  $-\text{CH}_2$ - increased the CMC



Fig. 2 Plot of CMC values versus alkyl chain length of alkyl *m*-xylene sulfonate

decreased, and every increased a  $-CH_2$ -, the CMC value was reduced by almost half (Fig. 2). For the Rosen reported equation of sodium alkane-1-sulfonate Log(CMC) =  $1.59 - 0.294 \times m$  [20].

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